State of California

AIR RESOURCES BOARD

Stationary Source Control Division

AIR MONITORING QUALITY ASSURANCE

VOLUME VI

QUALITY ASSURANCE IN THE TESTING OF STATIONARY SOURCES

By:

Engineering Evaluation Branch

January 1979

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QUALITY ASSURANCE IN THE TESTING OF STATIONARY SOURCES

6.1 INTRODUCTION

The source testing of stationary sources of air pollution is carried out to determine the types and amounts of pollutants emitted by these sources. Information gathered from source tests is used for planning, issuing permits, evaluating control systems, updating emissions inventories, and enforcing emissions limitations. For these purposes, source test data must be reliable; that is, the test must yield data in which users will have confidence. This reliability is provided for in a program called quality assurance.

Quality assurance is concerned with all the activities that have an impact, no matter how remote, on test results. Many of these activities are part of programs of quality control, the basic line or routine functions designed and implemented to provide a quality product. To these functions, quality assurance adds procedures that promote, review, analyze, audit, and report on quality control. In short, one way of looking at quality assurance is to see it as quality control of quality control.

The policies and procedures for achieving quality assurance in the testing of stationary sources are the topics of this volume. These policies and procedures are designed to produce test data that meet four criteria:

- Complete;
- Representative of the source's emissions;
- Accurate (that is, as near as possible to actual); and
- Precise (the agreement of repeated measurements of the same quantity; other words used to described a measurement's precision are repeatability, replicability, and reproducibility).

As used in source testing, the definitions of accurate and precise are derived from definitions used by systems engineers. According to these definitions, the meanings of the two words do not always overlap; that is, precise measurement techniques are not necessarily the same as accurate ones. An analogy that is sometimes made is of a cluster of shots in a target. If repeated shots form a tight pattern around the bull's-eye, the shooting is said to be accurate and precise. However, if repeated shots form a tight pattern in outer rings of the target, the shooting is said to be precise but not accurate. For more on the definitions of precise and accurate, the reader is invited to see the following: Environmental Protection Agency, Quality Assurance Handbook for Air Pollution Measurements, Volume I – Principles (Research Triangle Park, North Carolina: U.S. Environmental Protection Agency, 1976), pp. A17-A21; and E.J. Kovalcik, "Instrument and Measurement Errors," Test, April/May 1975, p. 10.

This volume has been designed to help program managers and source-test personnel gather data that meet the above criteria. Sections of the volume list measures and prerequisites planned to reduce the chance and magnitude of error. In addition, the volume prescribes steps to take to assure that the best possible results can be obtained with calibration gases, instruments, and other source-test equipment. Planning, auditing, and reporting on source tests are discussed, and appendices give specific methods for estimating the magnitude of errors, performing source tests, and maintaining and calibrating instruments.

6.2 GENERAL FACTORS INVOLVED IN STATIONARY SOURCE TESTING

6.2.1 GENERAL COMMENTS

The purpose of any source test program is to determine the pollutant concentration being emitted from a source. By measuring the pollutant concentration and the stack gas flow rate, the pollutant mass emissions can be calculated. The reliability of the test results depends largely on whether these measurements and all associated calculations are performed correctly.

To assure that a series of measurements represent the source's emissions, the measurements must be taken in a manner that is dependent on the process operation. Obtaining representative measurements is difficult in many cases. The difficulty arises because control agencies typically test a large variety of stationary sources and because some control agencies do not have sufficient resources to perform enough tests on the same source so as to establish a large data base. In addition, variations in the source operation during testing in some cases add a large degree of variability to source test data. When key parameters of a source operation can be monitored, it is sometimes possible to explain variations in test data. However, when source variations cannot be monitored, it becomes a serious problem to determine whether variations are due to process changes or errors in sampling techniques. Standard test procedures, knowledge of the source operation, and experience gained from testing must be used to decide if test results are indeed representative of the actual emissions.

6.2.2 ERRORS

The errors associated with source testing are generally described as <u>systematic</u> and <u>random errors</u>. A principle objective of a quality assurance program is to identify and reduce these errors.

Systematic errors are errors which can be reduced by the source test team. For example, systematic errors may result from:

1. Record keeping
2. System setup
3. Equipment maintenance and cleaning
4. Test personnel
5. Equipment calibration
6. Sample handling procedures

These sources of error, which are discussed further in later sections of this manual, can be reduced by the use of standard sampling and calibration procedures.

Random errors are more difficult to reduce. An example of an error is the uncertainty involved in reading an instrument. Repetitive readings of the same variable tend to decrease random errors. The significance of any random error may be small, but a series of random errors may be substantial in the calculated result. There are a number of statistical techniques available to determine the cumulative effect of random errors in the final result. Appendix A contains guidelines for estimating the significance of random errors.

6.3 TEST METHODS

Selection of the most appropriate method to be used for a specific test operation must take into consideration the chemical and physical properties of the gases to be tested. In addition, the types of analytical methods available for specific contaminants are also factors in determining the test method used. More specifically, the type of analytical method available may limit the test to integrated sampling, grab sampling, continuous sampling, or a combination of these. A look at problems associated with each method is presented here, and a description of the source test procedures themselves is given in Appendix B.

6.3.1 INTEGRATED SAMPLING

Integrated sampling can involve the passing of the exhaust gases through a filter, a chemical absorbing solution, water, or any combination of these throughout the test. The gas sample may also be collected in an evacuated container or pumped into a bag at a constant flow rate for the test duration. The filter, solution, and/or container contents are then analyzed in the laboratory. Use of this method results in average emissions being determined but cannot reveal any variations in emissions that may have occurred during the test. Almost all sampling for particulate matter is done by the integrated method.

To reduce the chances for error in obtaining a representative sample, the source test team must consider the isokinetic sampling rate and the probe location. (Isokinetic sampling adjusts the collection nozzle velocity to the same velocity as the stack to prevent alteration of flow streamlines).

Integrated sampling normally requires more handling of the test equipment, and the sample analyses techniques offer more opportunities for error than do most other methods. The use of a checklist will aid in reducing errors.

6.3.2 GRAB SAMPLING

A grab sample is a gaseous sample taken over a relatively short time period. Samples can be collected by (1) allowing the sample to fill an evacuated container, (2) inflating a flexible bag, (3) drawing the sample into a syringe, or (4) purging the air from a container with a gas sample. After collection, samples are taken to a laboratory for analysis.

Quality control problems associated with grab sampling are:

1. Contamination of container or bag. Assurances must be made that the material of the container does not react with the contaminant to be measured. Selection of the container material must be made considering the chemical properties and interactions with the stack gases. If containers are to be reused, they must be properly passivated with respect to the contaminant gas. A log of the types of samples collected in the container should be kept along with a record of passivation.

2. Loss of concentration of sample due to condensation on container walls. An absorbing solution or a heated container are some methods of controlling this loss.

3. Test results reflect only an instantaneous value. If the basic process has variations in emissions, many test samples may be required.

6.3.3 CONTINUOUS SAMPLING

Continuous sampling can be broken into two categories: extractive and in situ (in-stack) techniques.

Extractive sampling is the primary technique used by air pollution control agencies. It is accomplished by drawing a sample from the stack through a probe and a connecting line to electronic instruments that analyze the gases. Specific instruments must be approved by the responsible district. See Section 6.3.4 below for approval procedures. Variations in emissions and average emissions can be determined by this method.

Quality control problems associated with this technique are:

1. Conditioning of the gases before passing through the measuring device. Care must be exercised to assure that conditioning does not change the concentration of the specific pollutant to be measured.

2. Interference of components of the gas stream other than the specific pollutant to be measured. Interaction of pollutants while being measured must also be considered. Information can be obtained from the manufacturer of the instrument and/or by quality control testing in the laboratory.

3. Accuracy of calibration gases and possible interaction of the carrier gas. Quality control of calibration gases is covered in another section of this manual.

In situ sampling instruments measure concentrations as the stack gases flow by sensors located in the stack. This is usually a permanent installation, is not normally used by enforcement agencies for source testing and will not be discussed in detail. Quality control of this equipment can be accomplished by doing comparison testing with other approved sampling methods on a periodic basis.

6.3.4 ALTERNATIVE METHODS

When testing to determine compliance with a regulation, the test method specified in the regulation must be used. Where no test method is specified, a test method adopted by the ARB or by the responsible district must be used. If no appropriate adopted method exists, then the test method used must be approved by the regulatory agency and must be thoroughly documented.

If the organization performing the testing desires to use an alternative to the adopted test method, the organization must establish equivalence of the alternative test method in the following manner to the satisfaction of the responsible agency:

1. Details of the alternative method must be submitted to the district for review and discussion.

2. The laboratory analysis procedures must be described in the test method.

3. A minimum of three sets of parallel tests (with the adopted and the proposed alternative test method) must be performed on a single source type.

4. If possible, two of the sets of parallel tests should be performed during different source operating conditions.

When applying for permission to use alternative test methods, the applicant shall provide the responsible agency with a list of appropriate uses for the test method including the source operating conditions during which the proposed test method would be used.

Equivalency for continuous analyzers used for source sampling may be established if the analyzer satisfies the following criteria.

1. Operational and performance specifications equal or exceed those listed in Table 1.

2. The analyzer is capable of a 24-hour unadjusted continuous operation without the zero span or mean drift exceeding the limits specified in Table 1.

3. The analyzer's response has been verified by the use of side by side comparison testing with an approved analyzer or adopted method.

4. The analyzer's response is not significantly affected by other species present in the gas stream. This must be verified by performing interference tests.

Table 1
ANALYZER SPECIFICATIONS FOR SOURCE SAMPLING

	Typical Principle of Operation 1/	Typical Range or Ranges PPM	Minimum Detectable Sensitivity PPM	Noise Level % of Full Scale (peak to peak)	Response Time Time Interval from a step Change in Input Conc. at Inlet to Instrument Output Reading of 90% of Steady State	Zero Drift Change in Instrument Output After 24 hours of Unadjusted Continuous Operation Change % of Full Scale	Calibration or Span Drift in Instrument After 24 Hours of Unadjusted Continuous Opert. Change % of Full Sale	Precision Maximum Ave. Deviation From Mean Change % of Full Sale	Operating Temperature or Range	Linearity Maximum Deviation Between any Two Range Settings
Sulfur Dioxide	Photometric measurement of absorption of SO_2 radiation when subjected to ultraviolet light	0 – 300 0 – 3000	10	±1% Hi Range ±2% Low Range	< 10 secs.	< ±1.	± 2.0	± 2% on all scales	0 to 50°C	± 1
Oxides of Nitrogen	Photometric measurement of the chemiluminescence from the reaction of NO with ozone	0 – 25 0 – 100 0 – 250 0 – 1000 0 – 2500	2	±1%	< 10 secs.	< ±1%	± 2.0	± 2% on all scales	0 to 50°C	± 1
Hydrogen Sulfide	Photometric measurement of the chemiluminescence from the reaction of H_2S with ozone	0 – 5; 0 – 12.5 0 – 125 0 – 500 0 – 1250 0 – 5000	1	±1%	< 10 secs.	< ±1%	± 2.0	± 2% on all scales	0 to 50°C	± 1
Carbon Monoxide	Non-dispersive infra-red absorption	0 – 1000 0 – 10,000	10	±1%	< 60 secs.	< ±1%	± 2.0	± 2% on all scales	0 to 50°C	± 1
Carbon Dioxide	Non-dispersive infra-red absorption	0 – 5% 0 – 50%	1% of instrument scale	±1%	< 60 secs.	< ±1%	± 2.0	± 2% on all scales	0 to 50°C	± 1
Hydro-carbons	Flame ionization method of detection	0 – 10 0 – 100 0 – 1000 0 – 10,000 0 – 100,000	1 to 2 ppm Methane	±1%	< 10	< ±1%	± 2.0	± 2% on all scales	0 to 50°C	± 1
Oxygen	Uses the paramagnetic properties of oxygen	0 – 5% 0 – 10% 0 – 25%	.01% 100 ppm	±1%	< 60 secs.	< ±1%	± 2.0	± 2% of full scale	0 to 50°C	± 1

1/ Other types will also be acceptable.

6.4 PLANNING THE TEST PROGRAM

Planning of a test program, or even a single source test, must include all of the procedures and steps necessary to ensure that data obtained from the source tests will be accurate, precise, representative, and complete.

At the earliest opportunity, a pretest inspection of the source should be performed for the purpose of determining:

1. The type of process or operation. (Obtain a flow diagram if available.)

2. How the plant operating conditions are likely to affect emissions.

3. The types of emissions.

4. Where the emissions points are located, and if those points are accessible.

5. If the existing platforms, sampling ports, and power supply outlets are satisfactory.

6. The duct size, the temperature, pressure and velocity of the effluent gas stream, and an estimate of the moisture content of the effluent gases.

7. The type of location and condition of process instrumentation.

8. Safety hazards and special equipment required. (Obtain approval from appropriate safety officials if necessary.)

The gathering of such information may be facilitated by using a standard form such as the ARB's Engineering Investigation Report (See Appendix C).

The plan for collecting the sample requires preliminary knowledge of the process and its operating characteristics. The sample or test is useful as it relates to the process. The sampling plan should include full information on process feed rate, fuel rate, and stack gas velocity and temperature. It is essential to determine in advance if the process is steady state or cyclic. It is also essential to make arrangements with operating personnel to hold the process at normal operating conditions for the duration of the test, to operate at rated capacity, or to operate as specified in the operating permit.

If the process is cyclic, the sampling period should be planned to span at least one complete cycle. If the cycle is of short duration, it may be necessary to sample over several cycles to obtain an adequate sample. Cycles of extended duration may be broken into definable parts. It is often desirable to study parts of a cycle because an average obtained during each part may be more useful than an average over the entire cycle.

6.5 PREPARING FOR SOURCE SAMPLING

To minimize error care must be exercised in the preparation of the sampling trains and in the handling of samples.

6.5.1 GRAB SAMPLING

Two types of containers normally used for grab samples are flasks and flexible bags. Control must be exercised to assure that the containers are clean before use and that the sample will not deteriorate during transportation from the source to the laboratory.

6.5.1.1 Flexible Bags

Use only bags that are coated with Mylar, Tedlar, or other non-reactive material. No special preparation is required other than marking the bag for identification and assuring that the injection hole or valve does not leak. The best policy is to use a bag only once. However, it may be possible to reuse a bag for the same contaminant (with a separate log sheet for each bag). All bags should be purged several times with the sample gas before the sample is collected.

6.5.1.2 Flasks

Either glass or stainless steel flasks are used to prevent material reaction with the test sample. Glass flasks should be protected to prevent implosion or breakage. All flasks shall be thoroughly cleaned before use. Cleaning procedures are given in Appendix F.

Deterioration of test samples in transportation can be prevented by using absorbing solutions. If absorbing chemicals are required, only fresh reagents may be used, and these should be prepared by qualified laboratory personnel. In addition to the test sample flasks, a flask containing the absorbing solution is set aside for analysis as a control ("blank").

It is recommended that the flasks be evacuated at the sampling site rather than at the laboratory. Record the volume of flask, the volume of reagent, and the temperature and pressure of the flask. Identify each flask with time, date, location of the sample source, and identity of the person who collected the sample.

6.5.2 INTEGATED SAMPLING

6.5.2.1 Gaseous Samples

The flask or bag should be prepared as described in Section 6.5.1. Sampling probes should be cleaned as described in Appendix F. Connecting tubing should be a material that will not react with the contaminant to be sampled. If rubber is to be used as a connector, it should be visually inspected for cracking, or a leak check should be performed.

6.5.2.2 Particulate Matter Samples

Filters must be prepared according to the test procedure in Appendix B. All components must be thoroughly cleaned as described in Appendices B and F. Connecting tubing of various materials that do not interfere with particulate matter collection can be used. Care must be taken that no small particles are rubbed off when slipping the tubing onto the metal or glass connectors. Sharp edges on connectors should be filed, ground smooth, or fire polished to prevent this. Care must be exercised for glass to glass connections so that silicone-mating grease does not get into the sample train. It is suggested that grease not be used if possible. If used it should be used sparingly.

6.5.2.3 Leak Check

After assembly at the test site, the sampling train is checked for leaks by plugging the nozzle inlet and adjusting the vacuum to the maximum anticipated operating vacuum or 15 inches of mercury, whichever is greater. If a leakage rate (flow through the gas meter) greater than 0.02 cubic feet per minute is obtained, the leak must be found and repaired, and the leak check procedure must be repeated until satisfactory. Procedures for leak checking sampling trains are given in Appendix J. After the leak check, the vacuum should be released slowly by unplugging the nozzle before shutting off the pump. Failure to do so can cause a pressure surge through the train which may rupture the filter or displace reagents in the impingers. Reset the meter to zero or record the meter reading before sampling is begun.

6.5.3 CONTINUOUS SAMPLING

Equipment errors can be minimized by proper maintenance of the instruments and quality control of the calibration gases. Calibrations must be conducted both pre- and post-test, as well as periodically throughout the test. The sample conditioning systems of the instruments can also introduce errors. Care must be taken in setting up a sample conditioning system so that the conditioning does not interfere with the measurement of the contaminant or species of interest. The sample handling/conditioning system must be leak checked as in Section 6.5.2.3.

6.5.4 MISCELLANEOUS

All sampling equipment must be cleaned carefully to prevent sample contamination. Chromic acid cleaning solutions are recommended for cleaning glassware prior to beginning a test series. For metal analyses, certain types of glassware should not be used, and cleaning with nitric acid is recommended. The method description should include guidelines for choosing sample contacting materials.

A checklist will help assure that all test equipment being used has a valid calibration and is in good operating condition.

6.6 EQUIPMENT MAINTENANCE

Systematic errors arise from deficiencies inherent in the equipment or from inaccurate calibration. To minimize systematic errors, an effective maintenance and calibration program must be implemented. An equipment preventive maintenance program is described below, and the calibration program is presented in Section 6.7.

Preventive maintenance consists of those actions required to reduce equipment downtime, improve the reliability of source test results, and increase confidence in the results. The program consists of cleaning, lubricating, rebuilding, and adjusting equipment at predetermined intervals. A spare parts inventory should be maintained to assist in limiting equipment service time. A recommended spare parts list can usually be obtained from the manufacturer of each piece of equipment.

Preventive maintenance shall be performed on the following equipment:

1. Sampling pumps
2. Flow measuring devices (dry test meters, rotameters)
3. Pressure measuring devices (gauges)
4. Sampling instruments
5. Integrated sampling tanks
6. Mobile van sampling systems
7. Laboratory equipment (scales, etc.)

Other equipment may be added to the above list as required.

The service frequency shall be determined from experience and from manufacturer's recommendations. Each piece of equipment shall be identified by appropriate means, and each service should be recorded on a maintenance log sheet. Table 2 suggests acceptance limits and frequencies of maintenance for the equipment listed above.

ARB's procedures for scheduled maintenance and performance checks of mechanical test equipment are given in Appendix D. ARB's policy on preventive maintenance is to perform maintenance on a scheduled basis or whenever there are indications of performance deterioration or excessive wear.

The person in charge of the shop is responsible for:

1. Planning and implementing an equipment maintenance program
2. Ensuring that all maintenance and calibrations are performed on schedule
3. Maintaining equipment logs and keeping all service records up to date

The instructions for periodic maintenance, calibration, and special care of instruments and equipment are frequently updated. Abstracts from manufacturer's service and maintenance manuals are the primary sources for these updates. Also helpful is the assistance of factory representatives in initial installation and performance testing of the new instrument. And most important, manufacturer's instructions for operating an instrument must be followed to reduce error and prolong equipment life.

TABLE 2

MAINTENANCE SCHEDULE

EQUIPMENT	ACCEPTANCE LIMITS	FREQUENCY OF SERVICE	METHOD OF SERVICE
Pumps	1. Absence of Leaks 2. Ability to draw manuf. req. vacuum and flow	Every 500 hrs. of operation or 6 months whichever less	1. Visual insp. 2. Clean 3. Replace worn parts 4. Leak check.
Flow measuring device	1. Free mechanical movement 2. Absence of malfunction	Every 500 hrs. of operation or 6 months whichever is less After each test if used in H_2S sampling or other corrosive atmospheres	1. Visual insp. 2. Clean 3. Calibrate (see Sec.VI)
Sampling Instruments	1. Absence of malfunction 2. Proper response to zero, span gas	As required by manufacturer	As recommended by manufacturer
Integrated sampling tanks	1. Absence of leaks	Depends on nature of use.	1. Steam clean 2. Leak check
Mobile van sampling systems	1. Absence of leaks	Depends on nature of use.	1. Change filters 2. Change gas dryer 3. Leak check 4. Check for system contamination
Sampling Lines	Sample degradation less than one percent	After each test or test series	Flush with freon Blow filtered air thru line until dry

6.7 CALIBRATION

An effective program of equipment calibration is essential in assuring quality control of source test data. Virtually all source test measurement equipment requires periodic calibration checks of some kind. Since the specific equipment used by each agency varies, this section will include general guidelines for each type of equipment.

As a general rule, instruments and equipment used in source testing are calibrated at designated times and whenever major repairs are made. The calibration schedule, method of calibration, or standard of comparison and the acceptance limits for each analyzer are given in Table 3.

A record of each calibration is recorded in the instrument calibration logbook. This record must include the following information:

1. Instrument identification (serial or other identification number)

2. Date

3. Operation identification

4. Calibration technique

5. Description and identification of standard material used

6. Test or other code – for use in identifying samples for analysis by a referee (third-party calibration cross-checking)

7. Calibration curves, correction factors, etc.

8. Operator comments

6.7.1 CONTINUOUS ANALYZERS (EXTRACTIVE TYPE)

A variety of calibration techniques are available, but virtually all of them involve traceability to a known standard or to referee and analytical methods of well-established performance. For procedures used for establishing traceability of gases see Appendix E.

Two types of calibration are performed on all continuous extractive analytical instruments:

1. Primary (laboratory) calibration
2. Field calibration

6.7.1.1 Primary Calibration

In a primary calibration the response of the instrument is compared with a number of primary standard reference materials (SRMs). Primary SRMs are gases or other substances whose concentration, purity, and integrity are guaranteed by a recognized standard setting body such as the National Bureau of Standards (NBS).

The instrument's responses to five concentration levels within each working range are measured. Each response point is plotted on a graph. A calibration line (or curve) is constructed by drawing the line of best fit through the plotted points. This line should go through the origin of the graph. A test is applied to determine whether the instrument is within acceptable limits. For instruments with linear outputs, the largest deviation from the straight line may not exceed 2% of full scale at any point on the curve. The acceptable specification limits for each analyzer are given in Table 1. If the calibration curve is acceptable, that is, if it lies within the predetermined limits, the analyst can use it to translate instrument output into the appropriate concentrations. If the calibration curve is not acceptable, corrective action should be taken in accordance with the instrument's operating manual.

Changes within the instrument can cause the line of best fit to shift. Therefore, the instrument should be checked periodically by analyzing one of the SRMs used to construct the calibration curve.

6.7.2.2 Field Calibration

A field calibration consists of a functional check and a two-point calibration check (zero and span gas in expected concentration range). Field calibrations must be performed before starting and at the end of each test. During longer source tests, field calibration checks should be made periodically during the test and clearly marked on the recording chart. To increase the confidence of a two-point calibration, the linearity of the instrument should also be checked at least once during the test if possible.

6.7.2 OTHER SOURCE SAMPLING INSTRUMENTS AND EQUIPMENT

The recommended frequency of calibration and methods used for calibrating sampling instruments and equipment are shown in Table 3. A standard reference material traceable to the NBS should be used when available. If not, use calibration procedures recommended by the manufacturer, EPA, or ASTM. Where there are no reference materials for calibrating some instruments and devices, these devices should be calibrated against devices or instruments known to provide a higher degree of accuracy. Generally the calibration standard for mechanical equipment should be four to ten times more accurate than the equipment being calibrated.

TABLE 3

SAMPLING INSTRUMENTS AND EQUIPMENT CALIBRATION SCHEDULE

Instrument or Equipment Type	Frequency of Calibration	Standard of Comparison or Method of Calibration	Acceptance Limits
Orifice meter (large)	12 months	Calibrated Wet test meter	± 2% of volume measured
Orifice meter (small)	12 months	Bubble meter	± 2% of volume measured
Dry gas meter	12 months or when repaired	Wet test meter Bell Type spirometer	± 2% of volume measured
Roots gas meter	12 months or when repaired	Bell type spirometer	± 5% of volume measured
Turbine meter	12 months or when repaired	Bell type spirometer	± 2% of volume measured
S-Type pitot tube (for use with EPA type sampling train)	6 months	EPA Method 2 See 4.1:3	Cp constant > ±5% over working range Difference between the average Cp for each leg must be less than 2%
Vacuum gauges Pressure gauges	6 months	Manometer	± 3%
Field barometer	6 months	Mercury Barometer	± 0.2" Hg.
Temperature measuring	6 months	NBS Mercury Thermometer or NBS Calibrated Platinum RTD 1/	± 4^0F for Temp. <400^0F ± 1.5% for Temp. >400^0F
Temperature readout devices	6 months	Precision Potentiometer	± 2% Full Scale reading
Analytical balance	12 months	Should be performed by manufacturer or qualified laboratory	± 0.3 mg of stated weight
Probe nozzles	12 months	Nozzle diameter check Micrometer	Range > ±0.10mm for 3 measurements
Continuous analyzers	Depends on use frequency	As specified by manufacturers operating manuals, NBS Gases and/or EPA Ref. Methods	Satisfy all limits specified in performance specifications

1/ RTD – Resistance Temperature Device

6.8 QUALITY CONTROL IN CONDUCTING TESTS

The care with which the test team member makes instrument readings and performs various tasks controls the reliability of the data obtained from the test. Quality control procedures for different sampling methods are given here.

6.8.1 GRAB SAMPLING

In grab sampling, the probe or sample line is inserted into the stack and a portion of the flue gas stream is drawn through the sample line to purge all of the air from the system. A vacuum pump is used to draw the sample into a container or flask. For evacuated flasks, a vacuum gauge may be used between the valve and the flask to assure that the flask is still fully evacuated before the sample is taken. Multiple samples at each location must be taken to increase the reliability of the data.

6.8.2 INTEGRATED SAMPLING

6.8.2.1 Gaseous Samples

This method differs from grab sampling in that an orifice or restrictor is installed in sample line to control the amount of flow to the evacuated container or flask. Purging of the sample line as described under grab sampling should be accomplished.

6.8.2.2 Particulate Matter Samples

The sample rate must be proportional to the stack velocity, and to assure a representative sample the sample probe must traverse the entire stack cross section for most tests. The minimum number and location of sample points are to be as determined by Method 1 or from the test methods described in Appendix B.

To keep an isokinetic sampling rate, different rates of collecting the sample will be utilized when traversing the stack. The reading of the pressure sensing device and the condition of the pitot tube will affect the accuracy of the data. Frequent pitot tube calibrations and exact readings by the test personnel will reduce errors in this area.

After the test is completed and the final readings are recorded, a leak check is made of the sampling train. If the leakage rate is greater that 0.02 cubic feet per minute, discard test sample, correct the leak, and take a new sample.

To avoid contamination of the sample, cover the nozzle tip as soon as possible after the test run. As the sampling train is disassembled, all disconnected sections are also covered to prevent contamination. Extreme care must be exercised so that all sections of the sampling train are properly labeled for positive identification before transportation to the laboratory for analysis. It is recommended that one test team member be assigned for the responsibility of monitoring the test samples to protect them from loss, breakage, or contamination. Chain of custody procedures are used to preserve sample integrity while in transit to the laboratory and during the laboratory analysis. See section 6.8.5 below for more discussion on chain of custody.

6.8.3 CONTINUOUS SAMPLING

In continuous sampling a probe or sample is inserted into the stack; at a specific flow rate the gases are passed through a conditioner system and then to an electronic analyzer. The conditioner is normally designed to remove particulate matter and moisture but may also contain reagents to remove other gases that interfere with the specific contaminant being measured. Care must be exercised in adjusting the flow rate to the instrument during the test so that it is the same as was used during the pre-test calibration (zero and span). All necessary data must be recorded on a standardized form such as shown in Appendix G. Any shift between pre- and post-test calibration should be noted, along with any nonlinearity that may have been revealed in the periodic multipoint calibration of the instrument.

6.8.4 RECORDING OF DATA

All data relating to the operation of the sampling train must be carefully recorded on the field data sheet during the test. Any unusual occurrences in the process operation, unusual test instrument readings, or items that could possibly affect the test results should be noted on the data sheet. It is recommended that a checklist be used to assure all data needed for test calculations or process information are obtained.

6.8.5 CHAIN OF CUSTODY

For all types of testing, a chain of custody procedure must be used to assure that the sample analyzed is actually the sample taken under the conditions reported. Errors can be caused by delays in cleaning, contamination of the sample, and improper identification of the sample. The chances for error are greatly increased when too many people are permitted to handle samples. It is recommended that the responsibility for handling the samples be given to <u>one</u> person on the source test team and to <u>one</u> laboratory staff member. Ideally, a chemist would prepare the sample reagents or filter and then sign over these items to a test team technician, who will handle them at the test site, attach identification labels after testing, and return them to the same Chemist at the laboratory for analysis. A chain of custody form bears the names of the persons who prepared the reagents and the sampling train, recovered the sample, delivered the sample to the laboratory, and performed the laboratory analysis.

If the sample is not going to be analyzed immediately at the laboratory, a secure area must be used to store the sample.

6.9 POST-TEST QUALITY CONTROL PROCEDURES

Significant errors can occur when data are not reduced properly. Besides the sampling and analytical errors occurring during the test, human error can be introduced at any time between sample collection and data reporting.

6.9.1 INITIAL EXAMINATION OF DATA

As part of the post-test procedures, test data sheets should be examined to determine if any of the sheets contain any obviously abnormal values. Examine each abnormal value and correct whenever possible. However, the presence of an error may not be a sufficient reason for rejecting an entire test. Depending on the significance of the error a judgement can be made as to the acceptability of the results. A record should be maintained of test values which are judged to be invalid or otherwise suspicious.

6.9.2 DATA REDUCTION

Data reduction must take place with the aid of a standard calculation form or computer. The form should include step by step procedures required to obtain accurate final results. Computers are useful tools for handling large amounts of data and simplifying complex calculations and should be used whenever possible. All calculations and computer inputs must be checked by at least two people. Before the report is finished, the supervisor must make a final review and make the final decision of the validity of the data.

Data from continuous emission analyzers are recorded on strip charts. Strip chart recordings should be integrated with the use of a planimeter or similar device unless the recording is relatively constant with time. Data from strip charts must be checked by a supervisor.

6.9.3 VALIDATING DATA

Once the test results have been calculated, with adequate pre-cautions taken to assure accuracy, the next problem is determining if the results are representative of the actual emissions. Variability in results derived from multiple tests conducted on the same source at different times may be due to part or all of the following:

1. Variations in source operating parameters

2. Testing equipment and personnel variations both in the field and in the laboratory

3. Uncertainties inherent in the test method

Variations in the source operation may often be the most significant factor in the total variability of the test results. Knowledge of the source operation and monitoring of key operating parameters help to identify these variations. Variations caused by personnel can be minimized by operating a good training program. A good preventive maintenance program helps minimize variations in equipment operation.

The uncertainty of the results is an important aspect in determining the test's reliability. There are a number of statistical methods available for determining the uncertainty of testing results. Appendix A contains the derivation and sample calculations for one method.

Other useful tools for validating source test results are control charts that historically identify each test result with control limits to identify outliers (problem data).

When source test results fall outside of the control limits established by control chart calculations, the following questions should be asked:

1. Was the source operation varying?

2. Were the test data recorded and reduced properly?

3. Were equipment problems or probable human error encountered during the test?

4. Was the test method used appropriate for the source tested?

If the cause of the variations in results can be determined, the test report should note the problem, and another test should be conducted if necessary.

Reported results should, whenever possible, include the confidence interval of the results (for example, 120 ppm $\pm$10 ppm) for each run. The confidence interval can be obtained from the uncertainty analysis or from other statistically sound methods. The arithmetic mean should also be reported.

6.10 QUALITY ASSURANCE AUDITS

A quality assurance audit should be conducted by someone who is not a regular member of the source test team. This person will conduct performance audits and check source test results. Performance audits should be made periodically according to the organization’s needs. Performance audits fall into three distinct phases:

1. Sampling audits

2. Analysis audits

3. Data processing audits

6.10.1 SAMPLING AUDIT

A complete sampling audit should consist of simultaneous testing using different equipment and different operators. However, this type of audit is not always practical from a cost and personnel standpoint. In most cases, an acceptable sampling audit consists of a qualitative appraisal of the test procedures and test equipment used during the test. The auditor must verify that all equipment is functioning properly during the test and that all of the pre-test performance and calibration checks were made.

6.10.2 ANALYSIS AUDIT

An analysis audit consists of separate analyses of commonly collected integrated samples. Samples of known concentration can be submitted to two or more independent laboratories (interlaboratory testing). Samples can also be evaluated by separate chemists in the same laboratory (intralaboratory testing).

6.10.3 DATA PROCESSING AUDIT

Data processing audits consist of spot-checks of data reduction and calculation procedures. Computation methods are checked by inserting “dummy” data sets into computer programs and calculation sheets.

6.10.4 USE OF A CHECKLIST

Checklists may be used to assist in conducting audits. However, no checklist can cover all situations, so good judgment must be used by the auditor. If problems are found during any phase of a performance audit, the auditor should make every effort to locate and correct the problem. However, all problems must be recorded for later review.

A periodic review of the preventative maintenance program should be included as part of the audit process. This would consist of monitoring equipment downtime.

6.11 QUALITY ASSURANCE REPORTING

A quality assurance report to the manager of the source testing section should be periodically to outline the important aspects of the quality assurance program. The report should include pertinent facts in a short format.

The following items could be included:

1. System audit results

2. Performance audit results

3. Instrument or equipment operation

4. Percent voided samples versus total samples

5. Interlaboratory test results

6. Intralaboratory test results

7. Status of solutions to major problems

8. Recommendations for source test procedure changes

The format and content of the report will differ each for each organization depending on its source test program.

6.12 TRAINING

Personnel related errors arise due to carelessness, insufficient knowledge, or inadequate training. Many of these errors can be reduced by use of a training program for test personnel. The training should cover:

1. Operation and maintenance of equipment

2. Detection of electronic problems

3. Detection of sensor problems

4. Detection of mechanical problems

5. How to perform routine field repairs

6. How to calibrate instruments

7. Limitations of the instruments (range, responses, interference functions, temperature limits, moisture limitations, etc.)